PATENT SPECIFICATION (11) 1 498 031

1 498 031

(21) Application No. 36935/74 (22) Filed 22 Aug. 1974

(23) Complete Specification filed 6 Aug. 1975

(44) Complete Specification published 18 Jan. 1978

(51) INT. CL.² B04B 11/04

(52) Index at acceptance

B1D 154 164 260 332 334 338 351 356 372 375 381 385 604 639 686 687 691 692 693 696 702 705 712 714 715

(72) Inventor DONALD BOOTH

(54) LEVEL CONTROLLER

(71) We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, Imperial Chemical House, Millbank, London, SW1P 3JF, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the operation of centrifuges and to the detection and control of the level of slurry fed to centrifuges during their operation.

Centrifuges for separating liquid from solid may be designed either for continuous or batch operation. Batch centrifuges undergo a cycle of operations which may be repeated many times. The cycle includes the steps of

(a) feeding the slurry of liquid and solid,

(b) spinning to separate liquid and leave a solid 'cake', and

(c) removing the solid cake.

Adjacent steps may overlap, and additional steps may be included in the cycle, for example one or more cake washing steps and associated additional spinning steps. Moreover, the centrifuge may be run at different speeds at different stages of the cycle.

As is well known, straining batch centrifuges normally comprise a vessel known as the basket rotatable about the symmetric axis through the base. Such centrifuges may be operated with the axis of rotation either vertical or horizontal or, of course, in an intermediate position. During operation the slurry fed to the basket is forced by centrifugal force against the side, and forms an annulus. In what follows we shall refer to the smallest distance between the free surface of the annulus and the side of the basket at any point as the 'level' of the slurry.

The amount of slurry which can be fed to a batch centrifuge in any one cycle is limited by the fact that when the level in the basket exceeds a certain value, which depends on the geometry and the operation parameters, slurry starts to splash through or overflow through the opening provided for charging slurry and discharging solid cake. One method of ensuring that such loss of slurry does not occur is to feed a constant volume of slurry at each cycle, the amount fed being limited so that losses do not occur under any operating conditions. This is achieved in practice by feeding at a constant rate for a fixed time. Some separation of liquid from solid takes place during the feeding step, and with most slurries the rate of separation varies depending on the degree of choking or blinding of the screen lining the side of the basket which retains the solid and allows the liquid to pass through. In most cases it is necessary to interrupt the cycle of operations from time to time in order to clean the screen to ensure continued efficient separation. With a newly cleaned screen separation of liquid and solid is rapid, but the rate declines with continued operation until it is necessary to clean the screen again. When operating the centrifuge with a constant volume of feed per cycle, the amount fed must be such that at the slowest rate of separation, that is just before the screen is cleaned, there is no loss of slurry through the opening. When feeding this constant volume to a centrifuge with a newly cleaned screen, however, the condition of loss of slurry by splashing is avoided by a wider margin, and the basket would be capable of accepting a larger volume of slurry without loss. Thus the operation of a batch centrifuge with a constant volume of feed per cycle is inefficient in that its full capacity is not utilised.

A method has already been operated by which the capacity of the centrifuge is more fully utilised and in which the aim is to cut off the slurry feed only when the slurry level has reached a predetermined position. In this method the slurry level is detected by a float riding on the free surface of the slurry. The method usually employed combines the function of level detection with that of levelling the solid cake. In this method a cake leveller or snubber comprises a 'boat' which floats on the free surface of the slurry and is pivotally attached to a rigid stationary support, the boat trailing from the pivot in the direction of rotation of the centrifuge. As the level of slurry in the basket increases the boat will undergo angular motion round the pivot and arrangements are made so that when a predetermined level is reached this motion actuates control means (e.g. electric or pneumatic) which cut off the feed. For level detection this method suffers from disadvantages in practice. The boat is usually of relatively large dimensions and weight and requires a massive support which is difficult to position so that it supports the boat inside the rotating basket. Moreover, in the case of slurries which are non-homogeneous and contain, for example, lumps or aggregates of solid material, it is found that the boat will bounce on such lumps and so actuate the cut-off mechanism before the level of slurry reaches the predetermined value at which it was desired to cut off the feed. Moreover, similar bounce may occur due to standing waves on the free surface of the slurry. The result in either case is that inefficient use is made of the capacity of the centrifuge.

We have now found a method of level detection and control for straining batch centrifuges and apparatus therefor which obviate these disadvantages. Our invention provides apparatus comprising a straining batch centrifuge and means for detecting and controlling the level of slurry being fed to the basket of the centrifuge which means comprises a pipe not rotatable with the basket and adapted to be maintained filled with liquid, an open end of which pipe is positioned at a point in the basket to which the level is to be limited, and pressure-sensitive means connected to said pipe which on pressure rise in the pipe resulting when the slurry level reaches the open end of said pipe, is arranged to actuate control means to interrupt the slurry feed to the basket.

The pipe of our apparatus has an open end and is kept filled with liquid at least during the time when it is in use for level detection. The best means of achieving this is to have a continuous flow of liquid through the pipe, at least during the slurry feeding step of the centrifuge cycle. We have found that a pipe with an opening at the tip of internal diameter from 3 to 8 mm. is suitable, although our invention is not limited thereto, and with such an opening a flow of liquid of from 18 to 90 litres per hour is usually sufficient, such a quantity being a very small fraction of the volume of slurry passing through the centrifuge in the same time. The pipe itself, apart from the tip, may be of larger dimensions, and from the point of view of mechanical strength is desirably larger.

Our invention also provides a method for using the said apparatus which comprises continually feeding liquid to the pipe of the means for detecting the level of slurry so that the pipe is filled with liquid, rotating the basket and feeding slurry thereto until the level of slurry in the basket reaches the open end of the pipe when pressure rise in the pipe actuates the control means to interrupt the slurry feed to the basket, continuing rotation of the basket until a solid cake is obtained, discharging the filter cake from the basket, and repeating the slurry feeding and subsequent centrifuging operations.

For the purpose of level detection the nature of the liquid is immaterial. Since the liquid enters the slurry, however, it should preferably have no adverse effect on the slurry. Preferably the liquid should be miscible with the slurry liquid. Conveniently the liquid is the same as the slurry liquid. Where, as will often be the case, the slurry consists of solid and a liquid solvent from which the solid has been crystallised, so that the slurry liquid is solvent saturated with solid, it will be convenient to use the pure solvent as the liquid in the pipe of our apparatus. Water is, in general, a suitable liquid and so are organic solvents, for example, methanol, ethanol, isopropanol, acetone, acetic acid, benzene, toluene, xylene and chlorobenzene.

In order to conserve liquid and to prevent contamination of the solid cake with the liquid we prefer that the main flow of liquid through the pipe is limited to the period during which slurry is being fed to the centrifuge. However, in order to prevent blockage of the end of the pipe by the slurry we prefer that the main flow of liquid continues for a short period, say from 3 to 30 seconds, for example about 10 seconds, after the slurry feed has been interrupted. Moreover, particularly in the case of slurries comprising solid and saturated solvent, we prefer that there is a continuing subsidiary flow of liquid through the pipe during the periods when the main flow is interrupted, that is during

periods other than those in which slurry is being fed to the centrifuge. This is in order to prevent blockage of the pipe by crystallisation of solid material from solution adhering to the pipe. The subsidiary flow may be at a very slow rate, for example at a rate of approximately one tenth the rate of the main flow, e.g. at from 0.5 to 2.0 litres per hour. Liquid for the main and subsidiary flows may be supplied at the appropriate rates, for example by means of metering pumps or other metering devices.

For efficient operation we prefer that the tip at the open end of the pipe is disposed so that the plane through the open end of the tip is tangential to the free surface of the slurry at its point of contact therewith, or, more preferably, subtends a small angle, for example from 0° to 7°, with the plane which is tangential to the said free surface measured in the plane transverse to the basket axis and in the same rotational sense as that of the rotating said free surface. It is preferred to reduce the angle as the surface velocity of the slurry is increased. Moreover, we prefer to position the tip of the pipe at least half a revolution in the direction of travel of the basket periphery away from the point at which slurry is fed to the centrifuge basket.

When the slurry level reaches the open end of the pipe there is a considerable rise in pressure, for example to 3 bar. The rise in pressure associated with contact of the pipe with a lump of solid in the slurry is much less, and it is easy to select pressure-sensitive means which can distinguish these events.

The pressure-sensitive means may be any conventional means which operates on change of pressure, for example an electrical pressure switch or a pressure-sensitive pneumatic relay. The control means which is connected to the pressure-sensitive means and interrupts the slurry feed, may be any conventional control means, for example essentially electric, essentially pneumatic or essentially mechanical, which is capable of interrupting the slurry feed, in particular by closing a suitable placed valve.

By the use of the detection and control apparatus of our invention we are able to increase the capacity of a centrifuge to produce solid cake, compared with a system in which slurry is fed at a constant rate for a constant time, by as much as 20% or more. Compared with a system in which the slurry level is detected by means of a float, our apparatus is simpler and cheaper to construct and instal, is more consistent in operation, and with certain slurries leads to improved utilisation of the capacity of the centrifuge. Moreover, the critical part of the apparatus, namely the tip of the pipe, is exposed only to the liquid flowing through the pipe, except at the instant when the slurry level reaches it, and is thus only marginally exposed to any abrasive or corrosive influence which the slurry itself may have. Replacement of the tip at frequent intervals or the use of special alloys may therefore be avoided.

The apparatus of our invention may also be used to detect the need to clean the screen and to interrupt the normal cycle of operation and initiate the cleaning operation. The necessity to clean the screen arises from its gradual choking or blinding resulting in a progressively reduced rate of separation of liquid from solid. With automatically controlled centrifuges having a fixed interval for the spinning step, the effect of blinding of the screen will be a decrease in the solids content of the cake. The inclusion of higher proportions of mother liquor with the solid may lead ultimately to difficulties in handling the cake, an increase in the impurity level of the solid product and to process complications in downstream operations. It is therefore necessary to clean the screen before any of these effects reaches an undesirable level. The solids content of the cake can be assessed analytically, but this takes time and the level of undesirable effects may be passed before action is taken. Alternatively the solids content may be assessed empirically by an experienced operator, but if the operator is not attending to this particular task at the critical time, the level of undesirable effects may be passed before remedial action is taken. Moreover, owing to inconsistencies in the quality of the slurry coming forward for centrifuging and other factors, the lapse of time between a cleaning operation and the necessity for the next may vary so that it is only possible to clean according to a fixed time schedule by cleaning more often than necessity demands in many cases, with a consequent loss of centrifuging time and possibly excessive use of cleaning liquids.

The apparatus of our invention detects when a predetermined level of slurry is reached in the basket and at this point interrupts the slurry feed. With slurry fed at a constant rate this point will be reached in progressively shorter times as the screen becomes choked. Arrangement can therefore be made to time the feeding step and when the time falls below a predetermined value, corresponding with a cake consistency known to require cleaning of the screen, to interrupt the normal cycle at the appropriate point and initiate the cleaning operation. The appropriate point for interruption will normally be after the cake has been discharged from the basket. In one such

arrangement for use of our apparatus a fixed time interval from the commencement of feeding the slurry is signalled to the control system but the feed is prolonged until a cut-out signal is received on pressure rise in the pipe. When, however, the cut-out signal is received before the time interval signal, this situation can be detected and the washing operation initiated.

Thus our invention also provides, as a further feature, apparatus as hereinbefore defined which comprises in addition timing means for timing a fixed interval from the commencement of feeding the slurry to the basket and at the lapse of said interval sending a signal to discriminating means, discriminating means for receiving said signal from said timing means and a signal from the pressure-sensitive means on pressure rises in the pipe, and, on receipt of the latter signal before the former, for actuating control means to interrupt the centrifuging operation and initiate an operation for cleaning the centrifuge screen of the basket, following the centrifuging of the slurry batch whose feeding into the basket has resulted in said latter signal.

The timing means in this embodiment of our inventon may be any conventional apparatus for measuring lapsed time and for sending a signal after a predetenmined interval, especially a timing clock. It may be incorporated in the control system for timing other operations such as spinning. The discriminating means may be, for example, a double air-operated shuttle valve or two electrical switches coupled to latching relays. The control means may be any conventional control means, for example electrical, pneumatic or mechanical, for effecting the necessary operations.

Cleaning the screen may be effected by any suitable method. Washing the soreen with solvent is suitable in many cases.

Our invention will be better understood by reference to the accompanying drawings in which Figure 1 is an elevation in axial section of a centrifuge basket showing the slurry feedpipe and level-detecting pipe; Figure 2 is a sectional view perpendicular to the axis of the centrifuge along the line AA in Figure 1 in the direction of the arrows; and Figure 3 is a diagrammatic representation of the pressure sensitive device, and associated control means. In the drawings, 1 is the centrifuge basket, 2 is the centrifuge drive spindle, 3 is the lip of the basket opening, 4 is the slurry feed pipe, 5 is the level-detecting pipe and 6 the open tip thereof, 7 represents the maximum level of slurry in the basket and 8 represents the level of "solid" cake after spinning, 9 represents a pressure-sensitive pneumatic relay, 10 is a source of liquid under pressure for the level-detecting pipe 5, 11 is a flow meter which meters a continuous subsidiary flow of liquid to the pipe 5, 12 is a valve, actuated by a relay 13, which admits liquid to a flow meter 14 which intermittently meters a main flow of liquid to the pipe 5, 15 is the slurry feed valve actuated by a relay 16, 17 is a control unit receiving signals from the pneumatic relay 9 via channel 18 and sending signals to the valve relays 13 and 16 via channels 19 and 20 respectively. In operation the basket is continually rotating and the following sequence of events takes place:

(a) after discharging filter cake the slurry feed valve 15 and the valve 12 controlling the main flow of liquid to the level-detecting pipe are opened on signals from the control unit to the respective relays, and the centrifuge basket fills with slurry,

(b) on the level of slurry in the basket reaching the tip 6 of the pipe 5 pressure rise in the pipe actuates the pressure-sensitive pneumatic relay 9 which signals to the control unit which closes the slurry feed valve 15 and, after a predetermined delay, the valve 12 controlling the main flow of liquid to the pipe 5, via the respective relays, and the spinning operation then ensues during which the slurry at level 7 is reduced to solid cake at level 8, and

(c) the cake is discharged from the basket by means of a knife not shown in the drawings.

If desired one or more cake washing and associated spinning steps may be interposed between steps (b) and (c). The cycle of operations is repeated as often as desired.

Figure 4 of the drawings represents diagrammatically a suitable arrangement when the level-detecting pipe is used to detect the need to clean the centrifuge screen and to initiate the screen-washing operation. In Figure 4, in addition to the items shown which have already been mentioned, 21 is a double air-operated pneumatic shuttle valve, acting as discriminating means, receiving signals from the pressure-sensitive pneumatic relay 9 via channel 22 and from the control unit 17 via channel 23, and sending signals to the control unit via channel 24, 25 is the screen wash timer which receives signals from the control unit via channel 26 and which operates the valve 27 in the wash liquor line 28 by sending a signal to the relay 29 via channel 30 and which on completion of the screen wash cycle signals this event to the control unit via channel 31. In operation the following sequence of events takes place:—

(a) the slurry feed valve 15 opens on a signal from the control unit to relay 16,

the main flow of liquid in the level-detecting pipe 5 is established, and the centrifuge basket fills with slurry.

(b) after a predetermined interval the control unit sends a signal to the discriminating unit 21, whilst independently, on the level of slurry in the basket reaching the tip 6 of the pipe 5, pressure rise in the pipe actuates the pressure sensitive pneumatic relay 9 which signals firstly to the control unit which closes the slurry feed valve 15 and, after a predetermined delay, shuts off the main flow of liquid in the level-detecting pipe, and secondly signals to the discriminating unit, and then if the signal is received by the discriminating unit before the timing signal from the control unit the discriminating unit signals to the control unit to interpose event 'd' in the sequence of operations, and the spinning operation then ensues,

(c) the cake is discharged from the basket, and

(d) the control unit signals to the screen wash timer 25 which operates the screen wash valve 27 and allows wash liquor to flow in line 28 to wash the centrifuge screen for a predetermined time, and, after the washing operation is complete, signals to the control unit which reinstates operation 'a'.

Event 'd' is omitted in all cases where the appropriate initiating signal is not sent from the discriminating unit to the control unit.

The apparatus of our invention may be used in conjunction with any type of batch centrifuge and with any kind of slurry. It is particularly adapted, for example, to the control of centrifuging slurries of dimethyl terephthalate and methanol especially those obtained by the methanolysis of waste polyethylene terephthalate polymer. Owing to variations in the polymer, the latter slurries tend to be inconsistent, and may contain solid materials other than dimethyl terephthalate, for example carbonaceous materials or polymer delustrant such as titanium dioxide, which may tend to block the screen at variable and unpredictable rates. Smooth and efficient centrifuging of such slurries is achieved using the apparatus of our invention. With such slurries methanol is conveniently used for cleaning the screen. The apparatus is also suitable for controlling the centrifuging of slurries of terephthalic acid and acetic acid resulting from the catalytic air oxidation of p-xylene in acetic acid.

WHAT WE CLAIM IS:—

1. Apparatus comprising a straining batch centrifuge and means for detecting and controlling the level of slurry being fed to the basket of the batch centrifuge which means comprises a pipe not rotatable with the basket and adapted to be maintained filled with liquid, an open end of which pipe is positioned at a point in the basket to which the level is to be limited, and pressure-sensitive means connected to said pipe which, on pressure rise in the pipe resulting when the slurry level reaches the open end of said pipe, is arranged to actuate control means to interrupt the slurry feed to the basket.

2. Apparatus as claimed in Claim 1 in which the said open end of the pipe has an internal diameter at the tip of from 3 to 8 mm.

3. Apparatus as claimed in either of the preceding claims in which the pressure-sensitive means is an electrical switch.

4. Apparatus as claimed in either of Claims 1 or 2 in which the pressure-sensitive means is a pressure-sensitive pneumatic relay.

5. Apparatus as claimed in any one of the preceding claims in which the control means operates by closing a valve.

6. Apparatus as claimed in any one of the preceding claims which comprises in addition timing means for timing a fixed interval from the commencement of feeding the slurry to the basket and at the lapse of said interval sending a signal to discriminating means, discriminating means for receiving said signal from said timing means and a signal from the pressure-sensitive means on pressure rise in the pipe, and, on receipt of the latter signal before the former, for actuating control means to initiate an operation for cleaning the centrifuge screen of the basket, following the centrifuging of the slurry batch whose feeding into the basket has resulted in said latter signal.

7. Apparatus as claimed in Claim 6 in which the discriminating means is a double air-operated shuttle valve.

8. Apparatus as claimed in Claim 6 in which the discriminating means comprises two electrical switches coupled to latching relays.

9. Apparatus comprising a straining batch centrifuge and means for detecting and controlling the level of slurry being fed to the basket of the batch centrifuge substantially as herein described and illustrated with reference to the accompanying drawings.

10. A method of using the apparatus claimed in any one of Claims 1 to 9 which comprises continually feeding liquid to the pipe of the means for detecting the level of the slurry so that he pipe is filled with liquid, rotating the basket and feeding slurry thereto until the level of slurry in the basket reaches the open end of the pipe when pressure rise in the pipe activates the control means to interrupt the

slurry feed to the basket, continuing rotation of the basket until a solid filter cake is obtained, discharging the filter cake from the basket, and repeating the slurry feeding and subsequent centrifuging operations.

11. A method as claimed in Claim 10 in which the liquid filling the pipe is miscible with the slurry liquid.

12. A method as claimed in either of Claims 10 or 11 in which the slurry liquid is a solvent saturated with solid, and the liquid in the pipe is the said solvent in pure form.

13. A method as claimed in any one of Claims 10 to 12 in which the liquid in the pipe is water or an organic solvent.

14. A method as claimed in any one of Claims 10 to 13 in which the flow of liquid through the pipe is divided in time into a main flow in the period during which slurry is being fed to the centrifuge and a subsidiary flow during periods when the main flow is interrupted.

15. A modification of the method as claimed in Claim 14 in which the main flow of liquid continues for from 3 to 30 seconds after the slurry feed has been interrupted.

16. A method as claimed in any one of Claims 10 to 15 in which the tip at the open end of the pipe is disposed so that the plane through the open end of the tip is tangential to the free surface of the slurry at its point of contact therewith or subtends an angle of from 0° to 7° with the plane which is tangential to the said free surface measured in the plane transverse to the basket axis and in the same rotational sense as that of the rotating said free surface.

17. A method as claimed in any one of Claims 10 to 16 in which the tip of the pipe is positioned at least half a revolution in the direction of travel of the basket periphery away from the point at which slurry is fed to the centrifuge basket.

18. A method of using the apparatus claimed in any one of Claims 6 to 8 in which the centrifuge screen of the basket is cleaned by washing with solvent.

19. A method as claimed in any one of Claims 10 to 18 in which the slurry being centrifuged is essentially dimethyl terephthalate in methanol.

20. A method as claimed in any one of Claims 10 to 18 in which the slurry being centrifuged is essentially terephthalic acid in acetic acid.

21. A method for detecting and controlling the level of slurry being fed to a straining batch centrifuge substantially as herein described and illustrated especially with reference to the accompanying drawings.

Agent for the Applicants.
J. L. BETON.

Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd., Berwick-upon-Tweed, 1978.
Published at the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

1 498 031 COMPLETE SPECIFICATION
2 SHEETS This drawing is a reproduction of the Original on a reduced scale.
SHEET 1

FIG. 1.

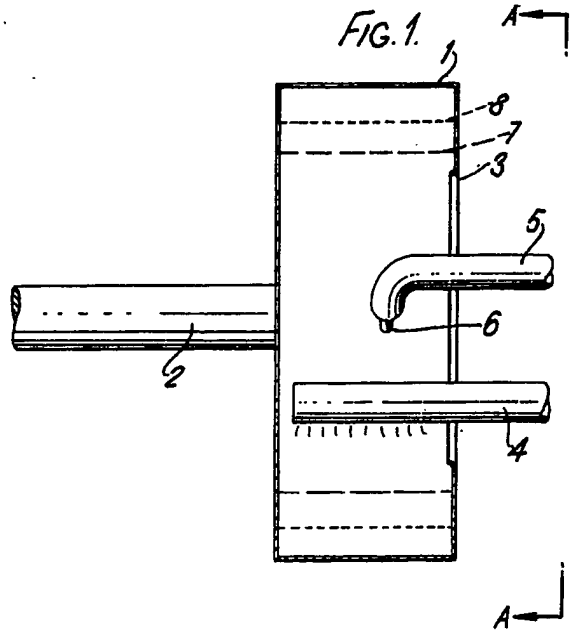
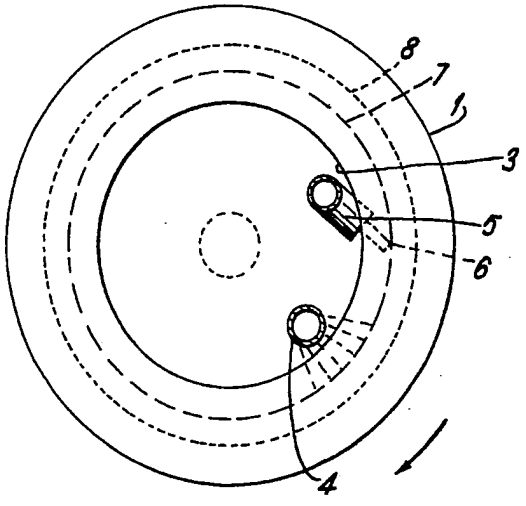


FIG. 2.



1 498 031 COMPLETE SPECIFICATION
2 SHEETS
This drawing is a reproduction of the Original on a reduced scale.
SHEET 2

FIG. 3.

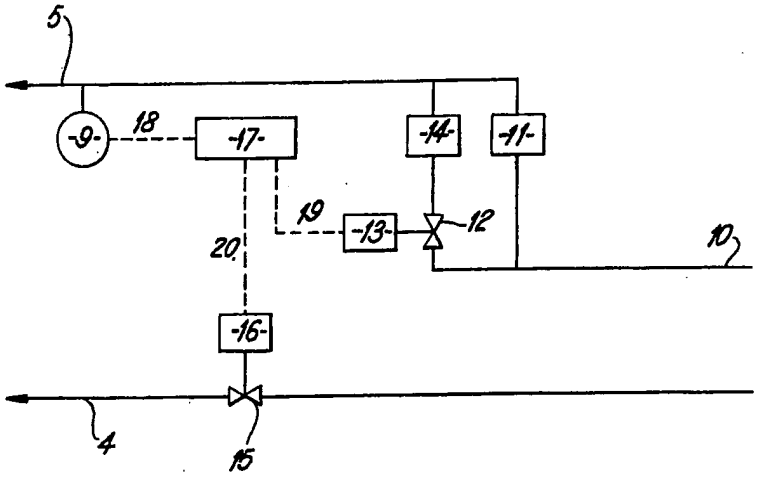


FIG. 4.